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Organic Audit Repository and Results of Performance Audits during Hazardous Waste Trial Burns and Compliance Source Tests

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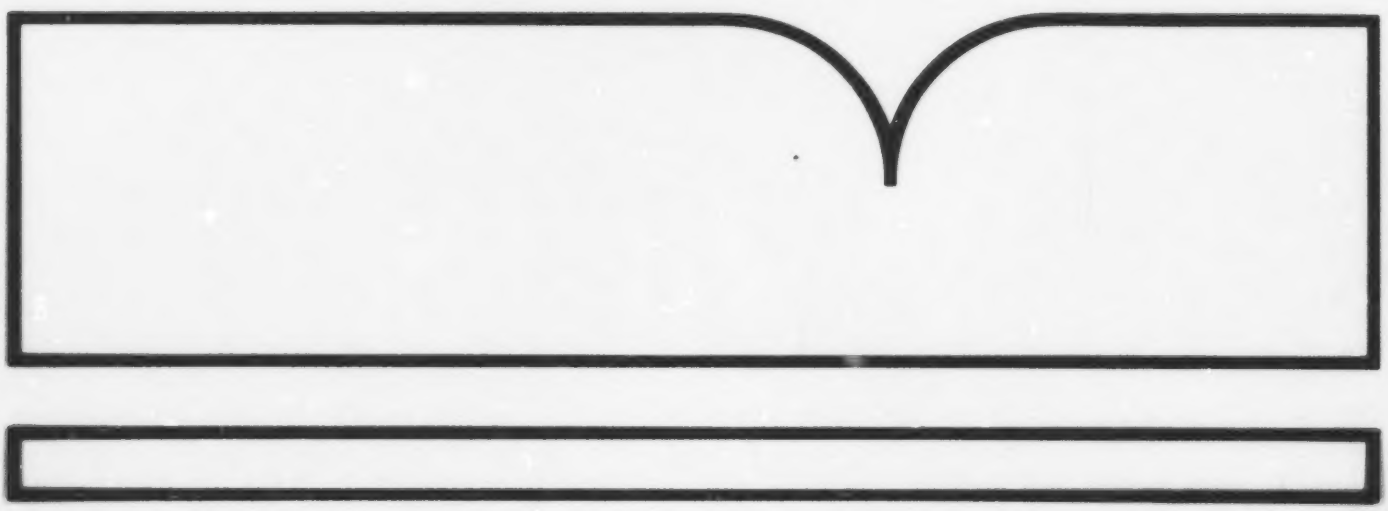

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ORGANIC AUDIT REPOSITORY AND RESULTS OF PERFORMANCE AUDITS DURING HAZARDOUS WASTE TRIAL BURNS AND COMPLIANCE SOURCE TESTS

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A repository has been established by EPA to provide audit materials to federal, state and local agencies or their contractors for use in performance audits to assess the accuracy of measurement methods used during (1) hazardous waste trial burns, (2) compliance source testing, and (3) ambient air measurements. This repository currently contains 21 gaseous organic compounds in five-, seven-, and nine-component mixtures at parts-per-billion (7 to 10,000 ppb) levels and 40 gaseous organic compounds at parts-per-million (5 to 700 ppm) levels in compressed gas cylinders.

Performance audits have been conducted using the repository gases to assess the accuracy of the measurement methods. Seventy-six and one hundred sixty-one performance audits to date have been initiated using ppb and ppm level audit gases, respectively. The audit results obtained with the ppb level component mixtures during hazardous waste trial burn tests were generally within ± 50% of the EPA concentrations. The audit results obtained with the ppm level organics during compliance source tests were generally within ± 15% of the EPA concentrations. The list of organics in the audit repository, stability data, and results of representative performance audits are described.

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ORGANIC AUDIT REPOSITORY AND RESULTS OF PERFORMANCE AUDITS DURING HAZARDOUS WASTE TRIAL BURNS AND COMPLIANCE SOURCE TESTS

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The measurement of air toxic organics requires sophisticated sampling and analysis systems. Agency personnel responsible for the use of air toxic measurements need to be concerned about the accuracy of such measurements. A repository has been established by EPA to provide audit materials to federal, state and local agencies or their contractors for use in performance audits to assess the accuracy of measurement methods used during (1) hazardous waste trial burns, (2) compliance source testing, and (3) ambient air measurements. This repository currently contains 21 gaseous organic compounds in five, seven, and nine component mixtures at parts-per-billion (7 to 10,000 ppb) levels and 40 gaseous organic compounds at parts-per-million (5 to 700 ppm) levels in compressed gas cylinders.

Studies of all gaseous organic compounds were performed to determine the stability of the compounds and the feasibility of using them as performance audit materials. Results indicate that all of the selected organic compounds were adequately stable for use as reliable audit materials.

Performance audits have been conducted using the repository gases to assess the accuracy of the measurement methods. Seventy-six and one hundred and sixty-one performance audits to date have been initiated using ppb and ppm level audit gases, respectively. The audit results obtained with the ppb level component mixtures during hazardous waste trial burn tests were generally within $\pm$ 50 percent of the RTI measured concentrations. The audit results obtained with the ppm level organics during compliance source tests were generally within $\pm$ 15 percent of the RTI measured concentrations. The list of organics in the audit repository, RTI measurement procedures, stability data, audit procedures and results of representative performance audits are described.

Introduction

The measurement of air toxic organics requires sophisticated sampling and analysis systems. Agency personnel responsible for the use of air toxic measurements need to be concerned about the accuracy of such measurements. The application of quality assurance practices is important to the generation of high quality measurement data. One such practice is the performance audit, which involves providing "unknown" or "blind" samples to laboratories for analysis. When conducted simultaneously with a source emission test, the performance audit provides an assessment of the measurement accuracy. Accuracy determinations are used in setting standards and in assessing compliance test results after emission standards have been promulgated. When it has been demonstrated that specific organic gases are stable enough to be used as performance audit materials, a performance audit should be included as an integral part of the promulgated test method.

The Quality Assurance Division of EPA's Environmental Monitoring Systems Laboratory has a program to supply accurate and stable audit materials for use when conducting source pollutant measurements. The Research Triangle Institute (RTI) operates a performance audit program for EPA utilizing an extensive repository of gaseous compounds. The repository currently contains 21 gaseous organic compounds in five, seven, and nine component mixtures at parts-per-billion (7 to 10,000 ppb) levels and 40 gaseous organic compounds at parts-per-million (5 to 700 ppm) levels in compressed gas cylinders. The balance gas for all gas mixtures is dry nitrogen. These cylinders are available at no charge to federal, state and local agencies or their contractors for use in performance audits to assess the accuracy of measurement methods used during (1) hazardous waste trial burns, (2) compliance source testing, and (3) ambient air measurements. A list of the gaseous organic compounds in ppm and ppb level audit repository are shown in Tables 1 and 2 respectively.

The gaseous compounds in Tables 1 and 2 are purchased in compressed gas cylinders from commercial suppliers. These cylinders, along with an appropriate delivery system, are used directly without dilution in the performance audits. The compressed gas cylinders are especially suitable as an audit material for a number of reasons - including simplicity, portability, stability, blind mode of operation (i.e. the user cannot readily ascertain the concentration levels), and ruggedness for interstate shipping.

Before being used as an audit material, the contents of each cylinder undergo a series of analyses at RTI. The cylinder contents are analyzed after initial receipt from the manufacturer to check the accuracy of the reported concentration of the test compound. The cylinder contents are then analyzed periodically over the course of a year and later on a yearly basis to estimate the compound stability. All concentration measurements are made using gas chromatography (GC) with flame ionization detection (FID) and/or electron capture detection (ECD).

Table 1 - Organic Gases in the PPM Level Repository.[a]

Acrolein	F-113[c]
Acrylonitrile	Hexane
Allyl chloride	Hydrogen sulfide
Benzene	Meta-xylene
1,3-Butadiene	Methane/ethane
Carbon disulfide	Methanol
Carbon tetrachloride	Methyl acetate
Carbonyl sulfide	Methyl chloroform
Chlorobenzene	Methylene chloride
Chloroform	Methyl ethyl ketone
Cyclohexane	Methyl isobutyl ketone
1,2-Dibromoethylene	Methyl mercaptan
1,2-Dichloroethane	Perchloroethylene
1,1-Dichloroethylene	Propane
1,2-Dichloropropane	Propylene
EPA Method 25	Propylene oxide
audit gas[b]	Toluene
Ethylene	Tetrachloroethane
Ethylene dibromide	Trichloroethylene
Ethylene oxide	Vinyl chloride

[a]The organic gases are in two concentration ranges. The low concentration range is between 5-50 ppm and the high concentration range is between 50-700 ppm.

[b]This audit gas is a mixture of an aliphatic, an aromatic, and carbon dioxide.

[c]F-113 is the compound 1,1,2-trichloro-1,2,2-trifluoroethane.

Table 2 - Organic Gases in the PPB Level Repository

Group	Concentration Range of Each Compound (ppb)
Group I*	7-90
	90-430
	430-10,000
Group II**	7-90
	90-430
Group III***	7-90
	90-430
Group IV****	7-90
	430-10,000

*Group I Compounds are carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride and benzene.

**Group II Compounds are trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane (F-11), dichlorodifluoromethane (F-12), bromomethane, methyl ethyl ketone, and 1,1,1-trichloroethane.

***Group III Compounds are vinylidene chloride, 1,1,2-trichloro-1,2,2-trifluoroethane (F-113), 1,2-dichloro 1,1,2,2-tetrafluoroethane (F-114), acetone, 1,4-dioxane, toluene, and chlorobenzene. Group III also contains pyridine but no certified concentration is currently available for this compound.

****Group IV audit cylinders are currently under development, and will be available about December, 1986. Group IV compounds are acrylonitrile, 1,3-butadiene, ethylene oxide, methylene chloride, propylene oxide and ortho-xylene.

Experimental Methods

Instrumentation

Analyses of ppm level audit gases are performed on a Perkin-Elmer Sigma 4 gas chromtograph with a flame ionization detector. Analyses of ppb level audit mixtures are performed on a Hewlett-Packard 5880A gas chromatograph equipped with flame ionization and electron capture detectors. The electron capture detector (ECD) is used principally for measurement of all the halogenated organics except vinyl chloride. A fixed volume of each gas in a sample loop is injected onto the GC packed column through a six-port gas sampling valve mounted near the injection port. After separation of the organics from nitrogen, a GC response is produced and electronically integrated. GC conditions for all analyses of ppm and ppb level audit gases have been previously described in two separate reports.[1,2]

Standardization and Measurement

Calibration of the gas chromatographs involves measurement of known concentrations of gases in nitrogen. The source or method of preparation of calibration standards varied depending on the gas involved. Calibration standards are prepared from pure compounds using pressure-dilution technique for most of the ppm level organic gases. National Bureau of Standards, Standard Reference Materials (NBS-SRM) of methane and propane are used for the calibration of the GC for the measurement of ppm levels of methane, ethane, propane, ethylene, and propylene. Permeation tubes purchased from commercial suppliers are generally used as calibration standards for all of the ppb level multicomponent organic mixtures. The permeation tubes are not used until their permeation rates, which are determined periodically by measured weight loss, have been well established. The permeation rate of each permeation tube is determined every 15 to 30 days by weight-loss determination.

A multipoint standard curve is generated for those compounds which exhibited a linear FID response. For those which show evidence of nonlinearity, at least two calibration standards are prepared each time a sample is analyzed. Both standards are prepared within 20 percent of the expected sample concentration.

Quality Control Procedures

Certain quality control procedures are followed. A set of primary standards for Group I, Group II or Group III ppb level multicomponent mixtures are analyzed each day depending on the type of sample gas mixture analyzed. These primary standards are analyzed as an internal quality control to verify the instrument calibration and the constancy of the detector response. The primary standards were prepared and analyzed by the National Bureau of Standards (NBS) as part of an EPA Interagency Agreement. A description of the NBS procedures for these standards has been reported.[3]

An example of RTI quality control analyses of these NBS primary standards is shown at the bottom of Table 5. Replicate injections of both audit cylinder gases and calibration standards are performed until no trends in the detector response are observed. Blank measurements are taken during cylinder analysis. Blank measurements have shown no signal above the baseline. Whenever calibration standards for ppm cylinders are prepared by pressure-dilution technique, the calibration mixture concentrations are verified with an independent method.[1]

Results and Discussion

Performance Audits

As stated previously, the USEPA (through RTI) supplies repository cylinders for audits upon request from the federal, state or local agencies or their contractors. The contractors must, however, be performing source emission tests on behalf of EPA or one of these other agencies to qualify for the performance audit. The performance audit should be conducted simultaneous with the actual planned source test. Performance audits prior to source tests to assess the proficiency of the measurement system (including the sampling and analytical personnel) are also encouraged.

When a request is received for an audit, the cylinder pressures are measured and the cylinder and glass manifold delivery system (for hazardous waste trial burn tests only) are shipped by overland carrier to the audit site. General instructions for conducting the audit are included with the cylinders. The audit results are reported to the agency (EPA, state or local) coordinator requesting the audit. There is no charge to the user except the cost of returning the audit cylinders.

To date, (January, 1986) 76 and 161 performance audits have been initiated using ppb and ppm level audit gases, respectively. Of these 76 ppb audits, 26 audits have been conducted to assess the accuracy of measurement methods used during hazardous waste trial burn tests. The results obtained for some of the ppb performance audits are shown in Table 3. The results of audits for the VOST methods are usually within the $\pm$ 50 percent limit stated in the VOST protocol.[4]

Five out of 40 gaseous organic compounds in the ppm repository have been found to be more popular for performance audits. These five compounds are: benzene, toluene, vinyl chloride, hydrogen sulfide, and the Method 25 gas mixture. Out of 161 performance audits initiated to date, 90 audits are conducted with these five compounds. The results for some of the ppm performance audits are shown in Table 4. Agreement of $\pm$ 15 percent with the audit concentrations measured by RTI is acceptable on ppm audits.

The principal results of these audits have been to assure that analyses are performed properly, to detect problems that could be corrected, and to document the accuracy of the measurement systems used during the hazardous waste trial burn tests or source compliance tests.

Stability Studies

The data collected over time from the measurement of cylinder concentrations are used to estimate the stabilities of the repository gases. Cylinder gas stability data are important for several reasons. First, the gaseous compounds in the cylinders must be stable to be considered by EPA as a reliable audit material. Second, if organic gases in the cylinders are stable, other investigators may more readily use cylinder gases as calibration standards and/or quality control check samples. Finally, if cylinder contents are stable, future EPA regulations may include performance audits as a means of assessing the accuracy of the measurement systems.

The term "stability" as it pertains to organic gaseous compounds, is defined here as the absence of change in concentrations over time for a given cylinder at a specified concentration level. The repository gases were analyzed initially by the manufacturer before shipment to RTI. These analyses were performed to demonstrate that the component(s) in the cylinders were stable at the manufacturers facility before shipment to RTI. The gases are then analyzed at RTI upon receipt from the specialty gas vendor to corroborate the vendor's analysis. The gas mixtures are again analyzed at 2 months, 6 months, 12 months, and annually thereafter following the initial RTI analysis to determine any change in the gas mixtures. The stability data obtained to date for all the ppb and ppm level organics are summarized and published in two separate reports.[1,2] An example of stability data for ppb organic gases in compressed gas cylinders are shown in Table 5.

An examination of the stability data for many of the organics in the ppb audit repository show that most varied by less than 10 percent over a two year period. The same is true for many of the organics in ppm repository over a 5-6 year period. The variation shown usually indicates the imprecision of the measurement process. The possible sources of experimental error that contributes to this variability include: (1) the variability of the sampling injection, (2) the instrumental variability in operating and detector parameters, and (3) variation in generation of calibration concentrations, and (4) lack of standard reference materials for many organics.

Summary and Recommendations

Cylinder gases of hydrocarbons, halocarbons, and sulfur containing organic species at the ppb and ppm levels have been used successfully as audit materials to assess the accuracy of measurement systems, especially

Table 3 - Performance Audit Results During Hazardous Waste Trial Burn Tests

Audit No.	Use of Audit Material	Audit Material	RTI Measured Conc. (ppb)	Client Audit Result bias(percent)[a]
5	VOST method evaluation prior to trial burn	Carbon Tetrachloride	11.0 ± 0.3	+31
		Chloroform	45.0 ± 0.9	+20
		Vinyl Chloride	20.5 ± 0.7	+ 7
28	VOST method evaluation prior to trial burn	Carbon Tetrachloride	9.5 ± 0.3	+22
		Chloroform	40.6 ± 0.9	+ 9
		Perchloroethylene	9.5 ± 0.2	+16
		Benzene	19.4 ± 0.6	+17
		Vinyl Chloride	20.6 ± 0.7	-13
26	GC method evaluation prior to trial burn	Trichlorofluoromethane	15.4 ± 0.5	-42
		Dichlorodifluoromethane	16.6 ± 0.7	-10
24	GC method evaluation prior to trial burn	Methyl Ethyl Ketone	19.4 ± 1.1	+18
		Trichloroethylene	14.7 ± 0.7	+16
		1,2-Dichloroethane	14.4 ± 0.7	+164
		1,1,1-Trichloroethane	14.5 ± 0.7	+110
29	VOST method evaluation prior to trial burn	Carbon Tetrachloride	11.4 ± 0.3	+ 5
		Chloroform	43.0 ± 0.9	+ 8
		Perchloroethylene	10.3 ± 0.2	+20
		Benzene	19.9 ± 0.6	+23
37	VOST method evaluation prior to trial burn	Acetonitrile	7.9 ± 0.6	+30
		Trichloroethylene	9.0 ± 0.4	+31
		Trichlorofluoromethane	9.4 ± 0.3	+23
		1,2-Dibromoethane	10.2 ± 0.4	+ 8
		1,1,1-Trichloroethane	10.1 ± 0.5	+ 5

[a] Client % Bias = 100 x $\frac{\text{Client Measured Conc. - RTI Measured Conc.}}{\text{RTI Measured Conc.}}$

Table 4 - Performance Audit Results During Compliance Source Tests

Audit No.	Industry/Source Tested	Audit Material	RTI Measured Conc. (ppm)	Client Audit Result bias(percent)[a]
39	Coke oven by product	Benzene in N_2 Benzene in N_2	8.20 85.4	- 2.6 - 8.7
79	Maleic anhydride Production	Benzene in N_2 Benzene in N_2	9.46 128	- 4.6 + 12.5
90	Oil shale	Hydrogen sulfide in N_2	437	- 3.6
113	Plastics	Vinyl Chloride in N_2	8.44	+ 10.2
143	Plastics	Vinyl Chloride in N_2 Vinyl Chloride in N_2	7.75 20.3	- 11.0 - 10.3
128	Surface coating	Method 25 gas in N_2 Method 25 gas in N_2	205 as C 1040 as C	+ 21.5 + 18.9
135	Polyester Resins Production	Method 25 gas in N_2	103.8 as C	+ 28.1
149	Plastic Foam Manufacturing	Methyl Ethyl Ketone in N_2 Method 25 gas in N_2	40.4 764 as C	- 24.8 - 9.3

[a] Client % Bias = 100 x $\frac{\text{Client Measured Conc. - RTI Measured Conc.}}{\text{RTI Measured Conc.}}$

Table 5 - Stability study for Group I Compounds
(Cylinder 1-A2)

	COMPOUND				
	Carbon tetrachloride	Chloroform	Perchloroethylene	Benzene	Vinyl Chloride
MANUFACTURER ANALYSIS					
Theoretical PPB	(9.5)	(39.6)	(9.4)	(18.9)	(20.0)
1st Analysis Date PPB	11/17/83 (7.7)	11/17/83 (42.1)	11/17/83 (9.2)	12/06/83 (15.8)	12/06/83 (18.3)
2nd Analysis Date PPB	12/16/83 (7.1)	12/16/83 (41.2)	12/16/83 (7.7)	12/15/83 (19.6)	12/15/83 (19.2)
RTI ANALYSIS					
1st RTI Analysis Date PPB	1/06/84 (9.3)	1/06/84 (40.0)	1/06/84 (9.2)	1/23/84 (19.6)	1/23/84 (20.2)
2nd RTI Analysis Date* PPB	59 (9.3)	59 (38.7)	59 (10.3)	49 (20.0)	49 (19.0)
3rd RTI Analysis Date PPB	239 (9.5)	239 (40.6)	239 (9.5)	226 (19.4)	226 (20.6)
4th RTI Analysis Date PPB	404 (9.9)	404 (37.1)	404 (9.1)	392 (19.4)	392 (20.4)
RTI QUALITY CONTROL**					
1st RTI Analysis Date PPB	1/06/84 (20.6)	1/06/84 (22.8)	1/06/84 (31.7)	1/23/84 (17.5)	1/23/84 (14.3)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)
2nd RTI Analysis Day PPB	59 (20.3)	59 (8.9)	59 (23.6)	49 (18.6)	49 (15.0)
NBS Concentration PPB	(19 ± 1)	(9 ± 1)	(22 ± 1)	(17 ± 1)	(15 ± 1)
3rd RTI Analysis Day PPB	238 (21.1)	238 (24.3)	238 (29.5)	226 (19.0)	226 (15.3)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)
4th RTI Analysis Day PPB	404 (20.6)	404 (23.2)	404 (28.7)	392 (18.1)	392 (14.1)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)

*Number of days after 1st RTI analysis date.

**NBS primary standard was analyzed as internal quality control on each day of RTI analysis. A different NBS primary standard was used on the 2nd RTI analysis for carbon tetrachloride, chloroform and perchloroethylene. RTI analysis concentrations are based on permeation tube standards.

those used during hazardous waste trial burn tests, compliance source tests, and ambient air monitoring.

Stability studies for many organic gases have been performed to determine the feasibility of using these materials in performance audits. Results indicate that many organic gases (see Table 1 and 2) are adequately stable to be used as reliable audit materials. However, six compounds (ethyl amine, paradichlorobenzene, cyclohexanone, 1,2-dibromoethylene, aniline and pyridine) are not recommended as audit materials due to either stability or analytical problems. Seventy-six and one hundred and sixty-one performance audits to date have been performed using ppb and ppm level audit gases, respectively. The audit results obtained with the ppb and ppm audit cylinders are generally within $\pm$ 50 percent and $\pm$ 15 percent, respectively of the RTI measured concentration.

Compressed cylinder gases of organics have been used successfully for performance audits to assess and document the accuracy of the measurement systems. It is recommended that a performance audit using compressed cylinder gas be conducted during each hazardous waste trial burn test and each compliance source test as a routine quality assurance procedure. Any federal, state, and local agencies and their contractors planning above mentioned tests may request a performance audit by contacting Mr. Robert L. Lampe, USEPA, Environmental Monitoring Systems Laboratory, Quality Assurance Division, Research Triangle Park, North Carolina 27711.

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